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IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF :  
OLEG STENZEL, ET AL. : EXAMINER: HANOR, S. L.  
SERIAL NO: 10/523,029 :  
FILED: SEPTEMBER 7, 2005 : GROUP ART UNIT: 1793  
FOR: HIGHLY DISPERSIBLE :  
PRECIPITATED SILICA

SECOND DECLARATION UNDER 37 C.F.R. § 1.132

COMMISSIONER FOR PATENTS  
ALEXANDRIA, VIRGINIA 22313

SIR:

I, André Wehmeier, declare and state as follows:

1. I am the same André Wehmeier who executed a Declaration under 37 C.F.R. § 1.132, with an execution date of May 27, 2008 (first Wehmeier Declaration) in the above-identified application.

2. I am familiar with the claims, and have read the Office Action mailed July 31, 2008, in the above-identified application.

3. The following experiments were conducted under my supervision and/or control.

4. New Examples 1A and 1B, which are according to the present invention, were prepared as follows:

Example 1A

80 l deionized water were introduced into a 120 l reactor with double shell heating and heated indirectly with steam until a temperature of 83°C was reached. Then, while stirring vigorously (shearing) at 83 °C for 100 minutes, 194.56 g/min water glass (density

1.346 kg/l, 27% SiO<sub>2</sub>, 8.00% Na<sub>2</sub>O) and approx. 49.77 g/min sulfuric acid (density 1.398 kg/l, 50.1% H<sub>2</sub>SO<sub>4</sub>) were added. This sulfuric acid was metered in such a way that a pH of 8.5 (measured at room temperature) prevails in the reaction medium. The reaction heat occurring during the reaction was dissipated via external cooling in order to maintain the reaction temperature.

After completion of the precipitation the addition of water glass was stopped and the sulfuric acid was added at 49.77 g/min until a pH of 3.0 (measured at room temperature (20°C)) was achieved.

The suspension that was obtained in this manner was filtered using a membrane filter press and rinsed with water. The filter cake was dried using a spin flash dryer.

The pulverulent product that was thus obtained had a BET surface area of 198 m<sup>2</sup>/g, a CTAB surface area of 193 m<sup>2</sup>/g, DBP absorption of 252 g/(100 g), and a Sears number V<sub>2</sub> of 13.5 ml/(5 g).

#### Example 1B

80 l deionized water were introduced into a 120 l reactor with double shell heating and heated indirectly with steam until a temperature of 68°C was reached. Then, while stirring vigorously (shearing) at 68°C for 100 minutes, 194.56 g/min water glass (density 1.346 kg/l, 27% SiO<sub>2</sub>, 8.00 % Na<sub>2</sub>O) and approx. 49.77 g/min sulfuric acid (density 1.398 kg/l, 50.1% H<sub>2</sub>SO<sub>4</sub>) were added. This sulfuric acid was metered in such a way that a pH of 8.5 (measured at room temperature) prevails in the reaction medium. The reaction heat occurring during the reaction was dissipated via external cooling in order to maintain the reaction temperature.

After completion of the precipitation the addition of water glass was stopped and the sulfuric acid was added at 49.77 g/min until a pH of 3.0 (measured at room temperature (20°C)) was achieved.

The suspension that was obtained in this manner was filtered using a membrane filter press and rinsed with water. The filter cake was dried using a spin flash dryer.

The pulverulent product that was thus obtained had a BET surface area of 272 m<sup>2</sup>/g, a CTAB surface area of 245 m<sup>2</sup>/g, DBP absorption of 243 g/(100 g), and a Sears number V<sub>2</sub> of 18.3 ml/(5 g).

5. As can be seen above, new Example 1A has a BET surface close to the lower limit of presently-pending Claim 1 of the above-identified application. The Sears number and the CTAB value are also close to the lower limit of the corresponding ranges of said Claim 1. New Example 1B, in contrast thereto, was designed as representative for a silica close to the upper limits of said Claim 1. BET, CTAB and Sears number of new Example 1B are close to the upper limits of said Claim 1. The respective values for BET, CTAB and Sears number as described in the specification herein for Example 1 are in between the two new Examples 1A and 1B.

6. The BET surface area, CTAB surface area, DBP number and Sears number for new Examples 1A and 1B, and Example 4 of U.S. 6,180,876 (Uhrlandt et al) which, in my opinion, is the silica disclosed in the prior art that is closest to the invention of presently-pending Claim 1, are shown in the following Table C:

Table C

		Example 4 <u>Uhrlandt et al</u>	New Example 1A	New Example 1B
BET surface area	m <sup>2</sup> /g	178	198	272
CTAB surface area	m <sup>2</sup> /g	156	193	245
DBP number	g/(100g)	282	252	243
Sears number V <sub>2</sub>	ml/(5g)	18.7	13.5	18.3

7. The silicas prepared by Examples 1A and 1B were then evaluated according to the same procedures as described in paragraphs 4 and 5 of the first Wehmeier Declaration, which paragraphs are hereby incorporated by reference.

8. Die C; 100°C (N/mm) for Examples 1A and 1B were measured according to paragraph 6 of the first Wehmeier Declaration, which paragraph is hereby incorporated by reference.

9. The high-temperature tearing resistance test was performed according to ASTM D 624 test (Die C). The test results are shown in following Table D:

Table D

	Example 4 <u>Uhrlandt et al</u>	New Example 1A	New Example 1B
Median Die C (ASTM D 624) 9 single values (100°C) N/mm	64	67	91

10. The comparison tests revealed that new Example 1A with

BET = 198 m<sup>2</sup>/g

CTAB = 193 m<sup>2</sup>/g

Sears number =13.5 ml/mg

shows the worst Median Die C value of all examples of the present invention. This Die C value, however, is still better than that of the closest prior art, i.e. Example 4 of Uhrlandt et al.

With increasing BET, CTAB and Sears number a trend can be seen. Accordingly, new Example 1B, which has the highest values for BET, CTAB and Sears number consequently shows the highest Die C value of 91, which is more than 42% better than Example 4 of Uhrlandt et al.

11. Besides a very balanced profile of rubber values, both new Examples 1A and 1B show that also in that very broad specific surface area range the improvement of high-temperature tearing resistance is given. This is of most interest for all high-performance, SUV, truck and also for motor bike tires in the tread compound as well as in all carcass compounds.

12. All three examples of the invention (Example 1 and new Examples 1A and 1B) show an improved high temperature tear resistance compared to Uhrlandt et al. Those silicas represent the whole range now claimed in the claims of the above-identified application and therefore prove, that the presently-claimed invention shows significant improvements compared to the closest prior art.

13. The Die C value for Example 4 of Uhrlandt et al was found to be 52 N/mm, under the experiment conducted for and as shown in the first Wehmeier Declaration, while the corresponding value herein, as shown in paragraph 9, was found to be 64 N/mm. The reason for the discrepancy is explained as follows.

14. Even if the same rubber is used, there are differences between the properties of different rubber lots. In addition, unavoidable variations of the mixing conditions have an influence on the physico-chemical properties of the rubber mixture. Even if the recipe is the same, variations of the test results are not unusual. Consequently, only data of one and the same test series can be compared directly and absolutely. In the above-described experiments of this Declaration, the median Die C value based on nine individual tests was used as the Die C value. In the first Wehmeier Declaration, only three single tests were conducted. For the above-described experiments of this Declaration, a new rework of Example 4 of Uhrlandt et al was used. It is not unusual that there is some variation between different reworks. As explained above, some variations of absolute values of Die C are usual between different test series. Nevertheless, within one test series, such as shown in the first Wehmeier Declaration,

the comparison of Die C values is still valid. Thus, even though the absolute values for Die C for Example 4 of Uhrlandt et al are different between this Declaration and the first Wehmeier Declaration, nevertheless, the data validly show improved Die C values compared to Uhrlandt et al.


15. As explained above, the absolute Die C value depends on the rubber mixture as well as the mixing conditions. Since different rubber mixtures are used for truck tires and bus tires, a minimum value of Die C cannot be defined.

16. The undersigned declarant declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

17. Further declarant saith not.

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13.01.2009  
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